Effective Reversed Phase thin Layer Chromatography for detection and separation of some metal ions from their aqueous solutions.

S. V. Choudhari[1], D.V. Bhagat[2]

Department of Chemistry, K.E.S Anandibai Pradhan Science College, Nagothane, University Of Mumbai

Abstract:

Reversed phase thin layer chromatography is an effective tool for separation of heavy metal ions ie Fe (III), Cu(II) ions from their two component mixture. The separation was carried out with thin layer of Silica gel-G as stationary phase using Acetic acid as mobile phase. Observed R_f values are helpful to determine the mobility of individual ions. Affecting factors like pH & concentration of acetic acid on R_f values of Fe (III) & Cu(II) ions was studied including determination of optimum conditions .

Key words: Reversed phase thin layer chromatography, Detection, R_f values, mobile phase, etc

1. Introduction:-

Reversed phase thin layer chromatographic technique used in identification and separation of metals ions from their aqueous solutions is effective an analytical technique. Now a days environmentalists stress on heavy metal ions removal from environmental samples, due to their toxicness that can be caused due to their bioaccumulative nature. Toxic levels of metal ions like Fe^{+3}, Cd^{+2} ,Ni^{+2} , Pb^{+2} ,Cu^{+2} , Zn^{+2} , etc .in water samples are harmful to human health .Currently several methods like Atomic absorption spectroscopy, Atomic emission spectroscopy, are available for detection of heavy metal ions, but This chromatographic technique is the most easier , low cost technique of separation of metal ions. So an objective of present studies includes detection and separation of the heavy metals from their aqueous mixtures and optimization of the conditions like pH, concentration.

2. EXPERIMENTAL SECTION

1. **Chemicals and Reagents**: Acetic acid , Silica gel - G ; Hydrochloric acid, sprayer for spraying reagents etc. .

2. **Stationary phase**- Silica gel plate is prepared with a glass plate of 4×2cm size (coated with silica gel and water), glass jar for the metal ions spot development on glass slides.

3. **Metal ions studied** – Fe (III), Cu (II).ions. Stock solutions of 0.05M $CuSO_4$. $.5 H_2O$ & of 0.05M $FeCl_3$ were prepared in the 0.1M Hydrochloric acid .

4. **Detecting regents** - 3% aqueous Potassium Ferro cyanide solution.

5. **Mobile phase**: -Aqueous solutions of Acetic acid.

3. Thin layer chromatography:

A. Preparation of Plates: Silica gel is mixed with distilled water in 1:3 ratio to obtain perfectly thick slurry. A glass plate is dipped in slurry and dried in oven for five minutes. Cool the plate at room temperature and this plate is used to perform thin layer chromatography.

B. Procedure- In experiment of thin layer chromatography, a drop of sample mixture is dropped at the bottom of the glass plate and dried .Now dried the plate is kept in a glass jar containing mobile phase solvent taking care that level of solvent remains below the drop of sample solution spot .

C. Separation & Identification of metal ions spots -The mobile phase solvent level strats ascending on the stationary phase due to capillary action, liquid filling the solid particle. Solutes particles get adsorbed on stationary phase depending upon its affinity towards the stationary phase. Spots of metal ions were identified with appropriate spraying agent ie 3% aqueous Potassium Ferro cyanide solution. The R_f values is calculated for each metal ion.

D. Optimization of conditions-To optimize the conditions for best use of this technique for detection of metal ions , and affecting factors are studied as given below in Table 1.

Table No-1-Variables under the study – pH, concentration

Variables			
Concentration	*0.01M*	*0.05M*	*0.1M*
pH	*1.3*	*3.3*	*4.9*

4. Result and Discussion:

From the studies of thin layer chromatographic experiments with acetic acid and silica gel as moble and stationary phases respectively for Fe,Cu binary system, following observations of Rf values are obtained and are given in Figure 1.
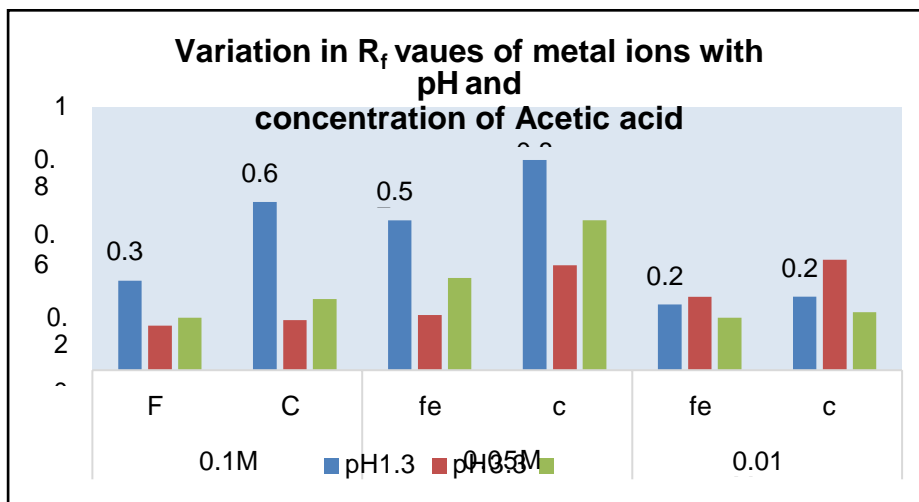

Figure 1-Observed variation in Rf values of Fe(III) & Cu(II) metal ions at pH 1.3, 3.3, 4.9 & concentrations 0.1M, 0.05M, 0.01M concentration of Acetic acid (mobile phase)

Similarly while optimizing conditions -1) pH 2) concentration

A. Effect of concentration-

Many experiments were performed at various concentrations of Acetic acid and variations in values of Rf of Cu^{+2} & Fe^{+3} ions are observed , are shown in Table 3.

Table 3 -Variation in Rf values of Cu^{+2} & Fe^{+3} ions **with different concentration.**

Adsorbent- Silica gel   **Mobile phase -Acetic acid**

Mobile phase	0.1MAcetic acid		0.05MAcetic acid		0.01MAcetic acid	
	Rf values		Rf values		Rf values	
Metal ions	Fe^{+3}	Cu^{+2}	Fe^{+3}	Cu^{+2}	Fe^{+3}	Cu^{+2}
pH=1.3	*0.34*	*0.64*	*0.57*	*0.80*	*0.25*	*0.28*
pH=3.3	*0.17*	*0.19*	*0.21*	*0.19*	*0.28*	*0.42*
pH=4.9	*0.20*	*0.27*	*0.35*	*0.29*	*0.20*	*0.22*

For the Fe+ Cu system, it is found that at 0.01 M Acetic acid solution, different metal ions showed Rf values closer to each other , hence separation of metal ions at this concentration is not possible. From the table3, it is revealed that at 0.05 M concentration of Acetic acid , metal ions showed little separation tendencies. The increase in Acetic acid concentration to 0.1M showed maximum difference in Rf values (0.30) of metal ions

Leading to satisfactory separation. Hence 0.1 M concentration could be fixed as optimum concentration and hence can be used for all further R_f measurements.

B) **Effect of pH**: Many experiments were performed at different pH .Variations in R_f values of Fe^{+3} & Cu^{+2}ions are observed and are shown in Table 4.

Table 4 - Variation in R_f values of Fe^{+3} & Cu^{+2}metal ions with change in pH . Adsorbent- Silica gel   Mobile phase -Acetic acid

Mobile phase	pH=1.3		pH=3.3		pH=4.9	
	R_f values		R_f values		R_f values	
Metal ions	Fe^{+3}	Cu^{+2}	Fe^{+3}	Cu^{+2}	Fe^{+3}	Cu^{+2}
0.1MAcetic acid	0.34	0.64	0.17	0.19	0.20	0.27
0.05MAcetic acid	0.57	0.80	0.21	0.19	0.35	0.29
0.01MAcetic acid	0.25	0.28	0.28	0.42	0.20	0.22

From experiments, effect of pH is studied for range of pH 1.3 to 4.9. From the observation Table 4 & Figure 1- it is evident that as concentration 0.1 M acetic acid is chosen as optimum concentration. At pH 1.3 , R_f values are of range in 0.34 - 0.64 ,but at pH 3.3 & 4.9 R_f values range from 0.17 to 0.35 .Thus it is seen that Fe^{+3} and Cu^{+2} metal ions move with solvent front at low pH value and showed maximum separation tendency at pH 1.3 which is chosen as optimum pH. At low pH, there are enough no. of H^+ ion to move the metal ion from the surface of stationary phase. There for Acetic acid gives better separation at low pH.

From above observation, the optimum conditions determined were as given in table 5

Table 5- Optimum conditions for the best use of Acetic acid as mobile phase

Mobile Phase	pH	Concentration
Acetic acid	1.3	0.1M

Many researchers like Jumde Meghna. (2015), Deshmukh L (2015), Sarang S Dhote*(2013),* Yeole, C. G(1983), Wanjari .S.M(2012) used silica gel as stationary phase for separation of heavy metals like Cr^{6+}, Fe^{3+}, Cu^{2+}, U^{6+}, V^{5+} , Ni^{2+} , Co^{2+} and Ag^+ ions etc in TLC technique and studied effect of pH and concentration of mobile phase . Similarly researchers used different mobile phases like 0.01–0.2 M aqueous sodium acetate (Deshmukh L et al, 2015), Mixture of Humic acid + DMSO at pH 4 (Jumde meghna et al , 2013) ,aqueous solutions of cationic and anionic surfactant(eg Sodium Dodecyl Sulphate) as mobile phase (Sarang S. Dhote.et al ,2013).for TLC technique . Also TLC can be applied for environmental samples like Tube Well Water (Deshmukh L et al, 2015), for separation of some rare earth metals.

5. Conclusion-

So, Thin Layer Chromatography is useful technique when used at its optimum conditions like concentration and pH , and have broader applications like heavy metal detection and separation ,impurity detection in food analysis and environmental analysis.

6.   References-

1. Deshmukh L., Kharat R. B.; "Separation of metal ions in tube well water sample by thin layer partition chromatography", Intern J. Environ. Analytical Chem.; Vol.36, 1988, 1-6.

2. Jumde Meghna.H, Gurnule Wasudev.B, "separation and identification of heavy metal ions by thin layer chromatography on silica gel-G", Journal of chemical and pharmaceutical Research, 2015,7(4):889- 895.

3   Sarang S Dhote, *et al*.: Separation of Various Metal Ions by Using Cationic Surfactant as A Mobil Phase By Thin Layer Chromatography International Journal of Chemistry.

4.   Yeole, C. G.; Shinde, V. M., "Reversed phase paper chromatographic studies of some rare earth elements", Analyst. 104, 1983, 1102-1107.

5. Wanjari .S.M, Deshmukh Lata, Paliwal .L.J " chromatographic separation of heavy metal cation on Silica gel -G with amino acid ,E- journal of chemistry, ,9(4),2446,2452,2012.